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VISCOSITY MEASUREMENTS OF SLAGS FROM COALS IN A PILOT-SCALE RESEARCH BOILER AND ASHES FROM POPLAR POWER GENERATING STATION

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by

D.H.H. Quon*, S.S.B. Wang* and T.T. Chen*

ABSTRACT

The flow characteristics of slags formed by burning of western Canadian coals were characterized by means of high temperature viscosity measurements. The results show that there is a general linear relationship between viscosities and SiO_2/Al_2O_3, $SiO_2/Al_2O_3+Fe_2O_3$ mass ratios and base/acid ratio of the slags. It can be concluded that the viscosities of the slags are sensitive to compositional variation.

Attempts were made to correlate the vicosities calculated by Watt and Fereday and Bonkomp's modified methods with the measured viscosities. The correlation is unsatisfactory. The computed viscosities are higher than the measured values.

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MESURE DE LA VISCOSITÉ DE LAITIERS DE CHARBONS DANS UNE CHAUDIÈRE DE RECHERCHE À L'ÉCHELLE PILOTE ET DES CENDRES DE LA CENTRALE DE POPLAR

par

D.H.H. Quon*, S.S.B. Wang* et T.T. Chen*

RÉSUMÉ

Les caractéristiques de fluage des laitiers provenant de la combustion de charbons de l'Ouest canadien ont été établies à partir de mesures de viscosité à haute température. Les résultats montrent qu'il existe une relation linéaire générale entre la viscosité d'une part et les rapport massiques SiO_2/Al_2O_3 et $SiO_2/Al_2O_3+Fe_2O_3$ et le rapport base/acide des laitiers d'autre part. On peut conclure que la viscosité des laitiers est fonction de la composition.

On a tenté en vain d'établir une relation entre les viscosités calculées par les méthodes modifiées de Watt et de Fereday et Bomkomp et les valeurs mesurées, les premières étant supérieures aux dernières.

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INTRODUCTION

Canadian utilities and industry are converting from use of premium liquid fuels to indigenous, often low-grade coals at a rapid rate in response to government initiatives to achieve oil independence and energy security. Canada has a vast quantity of coal deposits, and many newly developed thermal coals of unknown quality and performance require assessment prior to their commercial application. The Canadian Combustion Research Laboratory (CCRL) of CANMET has been actively involved in research on the performance of the Canadian coals in a pilot-scale boiler in collaboration with or in behalf of coal and utilities companies. The major objective of the work is to optimize conditions for achieving the best coal combustion in boilers. Ash fouling of the heat transfer surfaces is one of the most serious operating problems in burning low-grade coals. The ash deposits reduce the thermal efficiency and often lead to unscheduled shut-down of the boiler. Traditionally, the assessment of the slagging and fouling potential of the coals used in these pilot-scale experiments has been carried out using empirical indices based on the ash analyses of the raw coal, the analyses of the fireside deposits and visual examination of the deposits produced in the boiler. These indices include the ash fusion temperatures, base to acid ratio, slagging factor, fouling indicators, sodium content of the ash and calculated viscosity-temperature of the ash based on chemical composition (1,2). Viscosity measurements of ash deposits conducted in CANMET show that the calculated viscosity is higher than the measured value by 3 to 5 times (3,4). In order to understand the thermal behaviour of the ash deposits, it is necessary to examine the temperature-viscosity relationship of the deposits in detail and to develop a model that can be used for predicting the conditions under which slagging occurs.

EXPERIMENTAL

Two main areas are involved in this study:

(1) characterization of the phase compositions of the slags and their variation,

(2) experimental determination of the temperature-viscosity relationship of the slags.

MATERIALS

The boiler slags were produced by burning the western Canadian coals in a pilot-scale boiler at the CCRL of CANMET in 1982 and 1983. In addition, a sample each of bottom ash, fly ash and lagoon ash was obtained from Saskatchewan Power Corporation. These ashes were produced by combustion of lignite coal at the Poplar Power Generating Station, southern Saskatchewan. Chemical analyses of the slags and the ashes and their thermal behaviour are given in Tables 1 and 2 respectively.

Table 3 lists the samples and the types and origins of the parent coals. The slags were produced by burning high to medium volatile bituminous or subbituminous coals from Alberta and British Columbia, and the ash samples obtained from Saskatchewan Power Corporation were derived from a lignite coal.

CHARACTERIZATION

Prior to the viscosity measurements, the as-received slag samples were examined by X-ray diffraction (XRD) to determine the various phases present. A more detailed characterization was performed using scanning electron microscopy (SEM), electron microprobe analysis (EMP) and optical microscopy (OM).

A Leitz heating microscope was used to determine the melting behaviour of the slags.

Viscosity measurements were conducted for the slag and ash samples using a high temperature viscometer described in detail previously (3). Some of the slag samples supplied by CCRL weighed less than 40 g. The original viscometer set-up required a sample size of 130 g. As a result the viscosity measurement system was modified to accomodate the smaller samples. The viscometer bob was reduced from a diameter of 24 mm to 21 mm and from a height of 32 mm to 25 mm. The crucible for containing the melt was reduced from a capacity of 60 mL to 30 mL, with dimensions of 32 mm in diameter by 51 mm in height. The system was recalibrated using Brookfield silicone standard fluids: the calibration procedure has been described elsewhere (3). Figures 1 to 8 give the calibration plots between the viscosity and millivoltage output of the torque meter at the

various available rotational speeds.

The as-received slag samples were ground to minus 150 μm and calcined at 1000°C to reduce any volatiles that might have been present. These samples were pressed into small discs having a height of 1 cm by 2 cm diameter. The discs were then packed into the molybdenum crucible for melting.

RESULTS AND DISCUSSION

Megascopic and XRD examination of the slag samples indicate that the major phase in these samples is glass and there are minor variable amounts of mullite, cristobalite and quartz. Details of the phase characterization work will be given in a separate report (5).

There is no appreciable difference in thermal behaviour of the slags between the data supplied by the CCRL and those obtained by the Leitz heating microscope (Table 2). During the heating microscope work, it was observed that the samples tended to expand between the initial deformation and spherical softening temperatures. The volume expanded by approximately 50% of its original size. It appears that most of the as-received slag samples contain some trapped residual carbon. The expansion can be attributed to the internal generation of CO and CO_2 gas formed from this residual carbon. This volume expansion is undesirable and could create foaming problems during the viscosity measurements. Consequently, all the slag samples were calcined to 1000°C in air for an hour prior to their use in viscosity measurements. However, expansion was still observed during the viscosity measurement experiments. In some cases the volume expansion was so severe that part of the melt overflowed the top of the crucible, and in other cases occasional gas bubbles formed in the melt and interfered with the viscosity measurements.

Among the slag samples provided by CCRL, only eight were sufficiently large for viscosity measurements, and viscosity measurements for sample (A559-82) were abandoned because of the severe expansion of the slag. The viscosity versus temperature for the slags successfully measured are plotted in Fig. 9. In all cases, the viscosity decreases with increasing temperature. In general, the viscosity is higher for the slags derived from medium volatile bituminous coals than for those derived from the

subbituminous coals. The ashes derived from the lignite have the lowest viscosities.

EFFECT OF CHEMICAL COMPOSITIONS ON VISCOSITY

Previously, it was postulated that the viscosity of the slags is controlled to a great extent by the mass ratio of SiO_2/Al_2O_3 (3) and the results showed that the viscosity tends to increase with decreasing SiO_2/Al_2O_3 mass ratio.

The mass ratios SiO_2/Al_2O_3 for various slag samples were calculated and the results are given in Table 4. The viscosities at constant temperature for the different mass ratios of SiO_2/Al_2O_3 are plotted in Fig. 10. The results show that there is a general tendency for the viscosity to decrease with increasing SiO_2/Al_2O_3 mass ratio which agrees with the previous postulation.

It is well known that in silicate melts, Si^{4+} is regarded as an acid cation and acts as a network-former (in four-fold oxygen coordination), and basic cations such as Na, K, Ca, Mg, Fe^{2+} act as network-modifiers (in six-fold or higher coordination). Al^{3+} is amphoteric. As Fe^{3+} has the same charge and nearly the same ionic radius as Al^{3+} (0.051 nm versus 0.064 nm) and also exhibits isomorphic behaviour with Al^{3+} in some crystalline solids, it might be expected to behave analogously with Al^{3+} in the molten slag. The mass ratios $SiO_2/(Al_2O_3 + Fe_2O_3)$ were calculated and are given in Table 4. The mass ratios of $SiO_2/(Al_2O_3 + Fe_2O_3)$ versus experimental viscosities at constant temperature are plotted in Fig. 11. The results show that there is a general linear relationship between the log viscosity and mass ratio of $SiO_2/(Al_2O_3 + Fe_2O_3)$ for four of the slag samples. The viscosity decreases at constant temperature with an increase in $SiO_2/(Al_2O_3 + Fe_2O_3)$ mass ratio.

EFFECT OF BASE/ACID RATIO ON THE VISCOSITY OF SLAGS

The base to acid ratio (B/A) has been used extensively to predict the viscosities of metallurgical slags. This ratio is defined as follows:

$$B/A = (Fe_2O_3 + CaO + MgO + Na_2O + K_2O)/(SiO_2 + Al_2O_3 + TiO_2)$$

where each oxide is expressed in weight per cent of total slag. The B/A ratio was also used to predict the viscosity of the coal ash. It was reported that there is a general correlation between B/A ratio and the viscosity of the coal ash (1,2) and that the viscosity decreases as the B/A ratio increases to 1. The B/A ratios of the slag samples were calculated and are given in Table 5. These ratios are plotted against measured viscosities at a constant temperature in Fig. 12. The results show that, for certain slag samples, viscosity and B/A ratio correlated well as indicated by the almost linear relationship between these two parameters.

ESTIMATION OF VISCOSITY OF SLAGS

Based on experimental measurements on ashes of more than 100 British coals, Watt and Fereday proposed the following viscosity-temperature-chemical composition relationship (6).

$$\log \eta = \frac{10^7 M}{(T-150)^2} + C \qquad \text{.....(1)}$$

where

η = viscosity in poises

T = Temperature, °C

$M = 0.00835(SiO_2)+0.00601(Al_2O_3)-0.109$

$C = 0.0415(SiO_2)+0.0192(Al_2O_3)+0.0276(Fe_2O_3)+0.016(CaO)-3.92$

The oxides in parentheses are the concentrations of these oxides in the melt expressed in weight per cent.

Using equation (1), the viscosities of the present slag samples were calculated and are compared with the measured viscosities ofthese slags in Fig. 13. The calculated viscosities are higher than the measured ones at any given temperature for most slag samples. Only for sample A601-82 do the calculated and the experimental values agree reasonably well. Similar disagreement between the calculated and experimental viscosity data was reported by others (7,8). To improve the accuracy of viscosity prediction, Bonkamp (8) modified the Watt and Fereday equation for calculating constants M and C to account for the MgO content. These two constants are:

$$M = 0.0104291(SiO_2)+0.0100297(Al_2O_3)-0.296285$$

$$C = 0.0154148(SiO_2)-0.0388047(Al_2O_3)-0.0167264(Fe_3O_4)-0.0089096(CaO)-0.012932(MgO)+1.04678$$

Fig. 13 gives the viscosities of four slag samples calculated according to the Bomkamp formula. The results show that the calculated viscosities are appreciably higher than those using the Watt and Fereday method.

Coal slag is a complex material, consisting primarily of silica, alumina, iron oxide and lime. A minor change of the major constituents or of some of the minor consistituents such as alkalies and alkali earths, and the variation of the oxidation state of the iron between the ferrous or ferric state would alter the fluid characteristics of the slag. It is not surprising that the calculated viscosities for a particular slag are in disagreement with the experimental values as these results are computed based on the major constituents present in the slag. A small difference in composition may have a great influence on the viscosity of the slag. In their study of the viscosity of coal ashes, Schobert et al. (7) reported a difference in temperature-viscosity relationship for two ash samples having similar chemical compositions. The two samples differed only by 0.6 mass per cent in Na_2O, a minor constituent, and by no more than 2.0 mass per cent in the major constituents silica, alumina, ferric oxide and calcium oxide. This indicates that minor changes in composition can significantly affect viscosity, and may explain in part that a precise prediction of viscosity based only on the major constituents in the slag sample is difficult. As the calculated viscosity is based on the composition of the slag, it is necessary to examine the slag compositions after being used for viscosity measurements to determine whether compositional change has taken place due to the loss of volatiles such as alkalies and alkali earths. Two samples of solidified melt from the viscosity runs were submitted for chemical analysis. The results obtained are given in Table 6 in comparison with the chemical composition of the original as-received slag samples. As noted, the compositions of the melts differ only slightly from those of the as-received slag samples.

SLAGGING FACTOR

The slagging factor of the coal ash can be calculated based on the thermal behaviour of the ash. This factor is expressed as (1):

$$Ts = \frac{\text{(hemispherical temp. + 4 * initial deformation temp.)}}{5}$$

where the temperature is expressed in °F.

To calculate the slagging factor, the thermal behaviour data of the slag samples given in Table 2 were used. The severity of slagging is classified according to the suggested rating scheme (1) given in Table 7. The computed slagging factors for the various slags are given in Table 8. It appears that the slagging potential for the slags increases with decreasing viscosity at a constant temperature.

CONCLUSIONS

The primary objective of this study was to provide direct support to determination of the cause of slagging in a pilot-scale coal fired boiler using the viscosity measurement technique. At this stage of the work, several observations have emerged:

(1) There is a fair correlation between viscosity versus SiO_2/Al_2O_3, $SiO_2/(Al_2O_3+Fe_2O_3)$ and B/A mass ratios for certain slag samples. This indicates that the major constituents may be used in a qualitative way to predict the viscosity-temperature relationship of a slag.

(2) Both Watt and Fereday's method and Bomkamp's modification used for viscosity-temperature calculation produce higher values than those determined experimentally.

With regard to viscosity measurements, further research is needed. Particular attention should be directed toward the study of the viscosity modification due to the compositional variation of the minor constituents such as alkali and alkali earths as well as the oxidation state of the iron. Undoubtedly the minor constituents are playing a important role in the structural modification of the melt.

With the recognition that current empirical tests to predict the high temperature behaviour of coal slag have limited usefulness, and that measurement of slag viscosity is a tedious task, it is desirable to be able to predict the viscosity-temperature relationship from other data that is readily available. To develop a suitable formula for calculating the viscosity-temperature relationship for different slags derived from Canadian coals, much work is required to be done for coal ashes and slags of different compositions.

ACKNOWLEDGEMENTS

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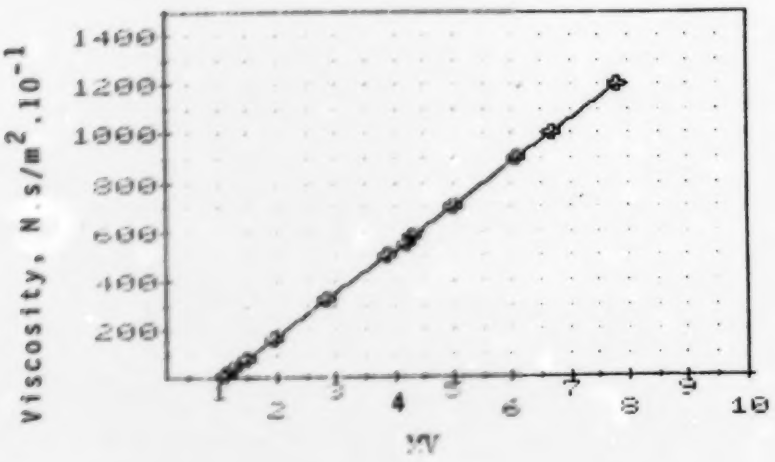

Fig. 1 - Calibration of viscosity versus millivolts at a spindle rotation rate of 0.5 rpm.

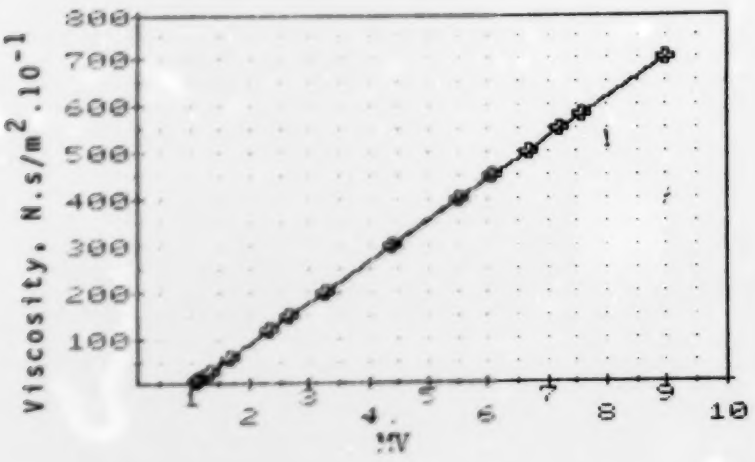

Fig. 2 - Calibration of viscosity versus millivolts at a spindle rotation rate of 1.0 rpm.

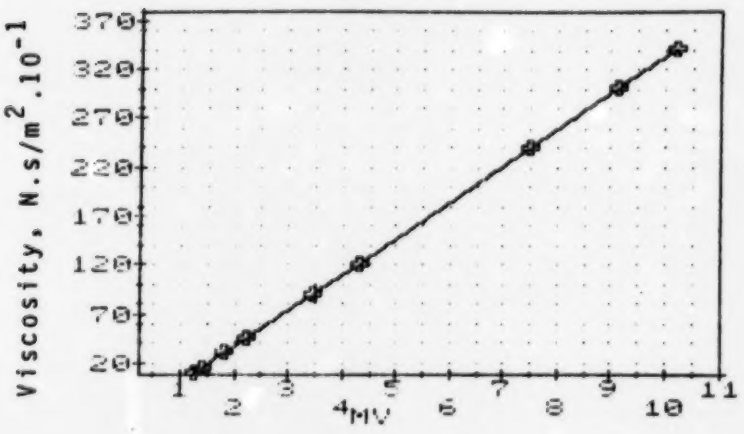

Fig. 3 - Calibration of viscosity versus millivolts at a spindle rotation rate of 2.5 rpm.

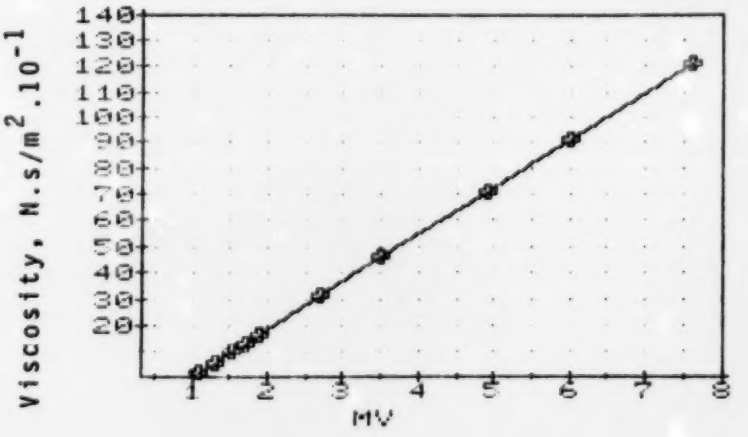

Fig. 4 - Calibration of viscosity versus millivolts of a spindle rotation rate of 5 rpm.

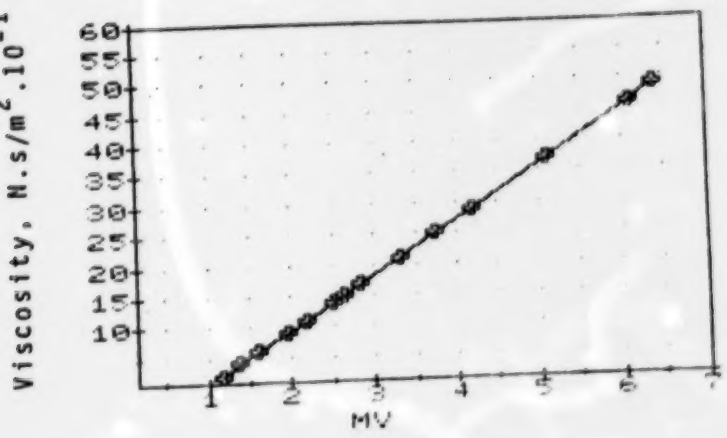

Fig. 5 - Calibration of viscosity versus millivolts at a spindle rotation rate of 10 rpm.

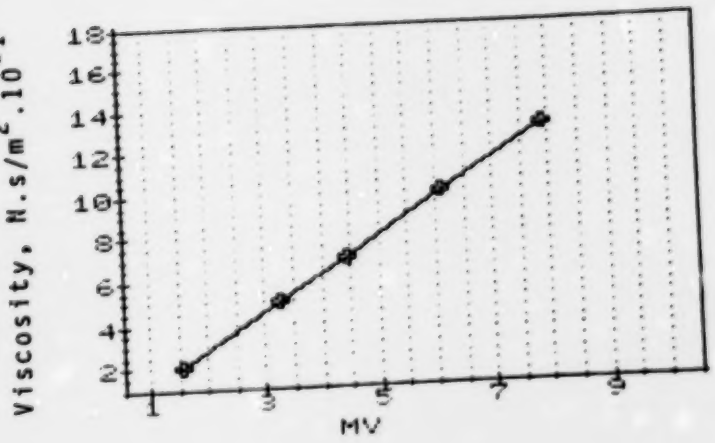

Fig. 6 - Calibration of viscosity versus millivolts at spindle rotation rate of 20 rpm.

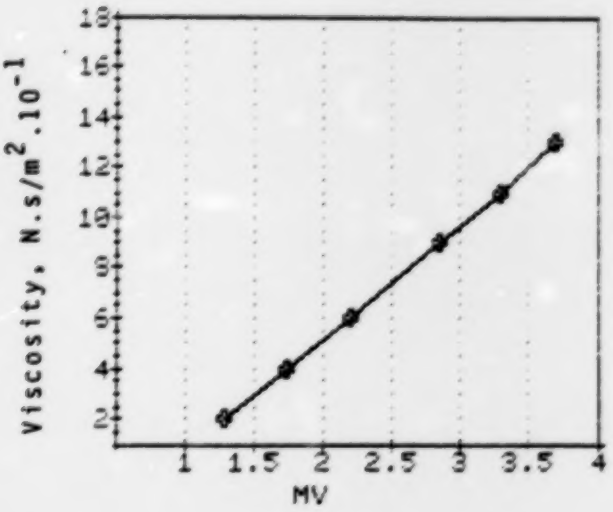

Fig. 7 - Calibration of viscosity versus millivolts at a spindle rotation rate of 50 rpm.

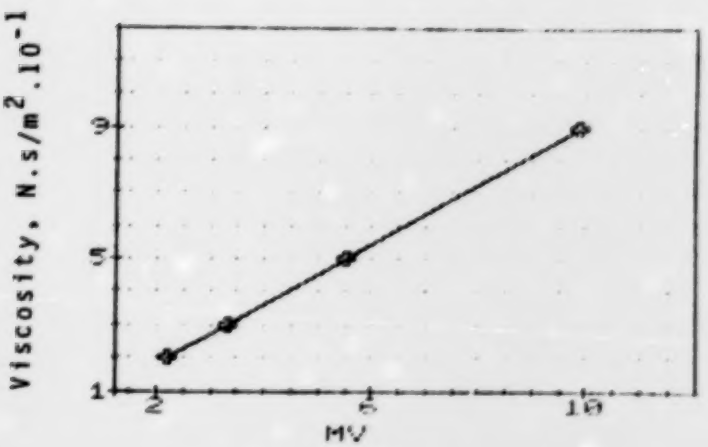

Fig. 8 - Calibration of viscosity versus millivolts at a spindle rotation rate of 100 rpm.

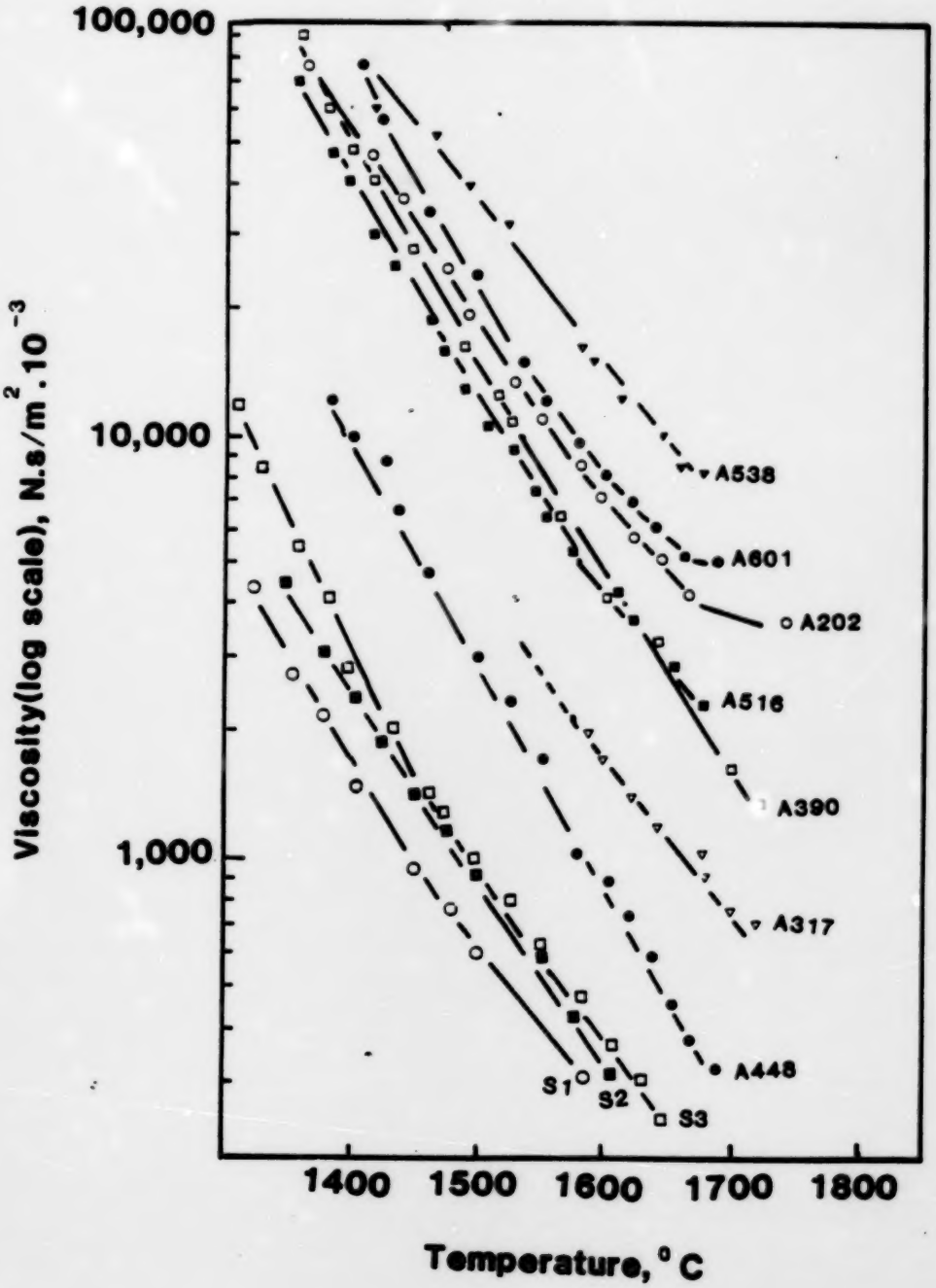

Fig. 9 - Viscosity as a function of temperature for the slag samples.

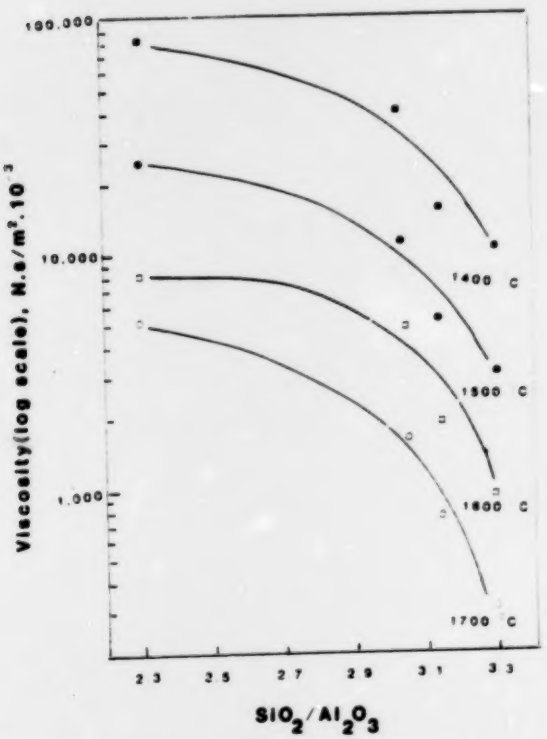

Fig. 10 - Viscosity as a function of SiO_2/Al_2O_3 mass ratio at constant temperature.

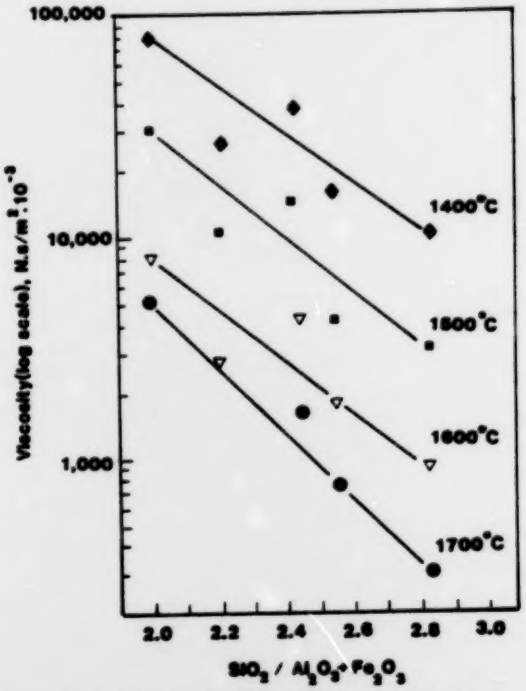

Fig. 11 - Viscosity as a function of $SiO_2/(Al_2O_3+Fe_2O_3)$ mass ratio at constant temperature.

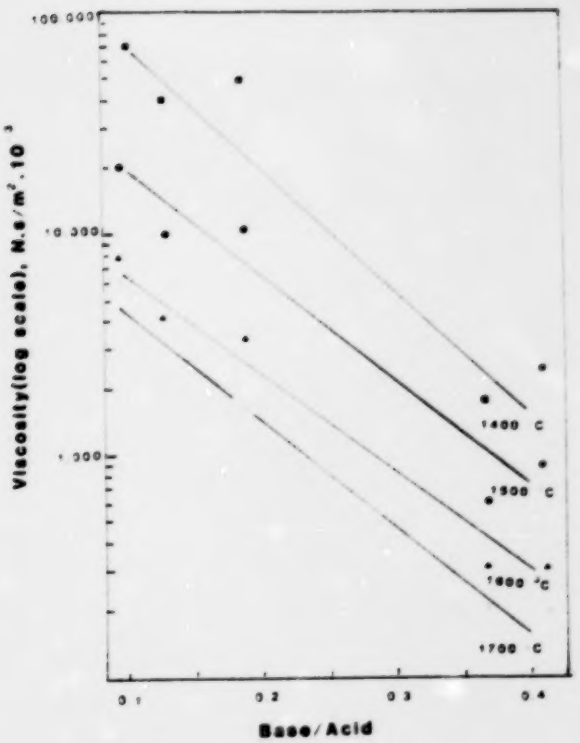

Fig. 12 - Viscosity as a function of B/A ratio at constant temperature.

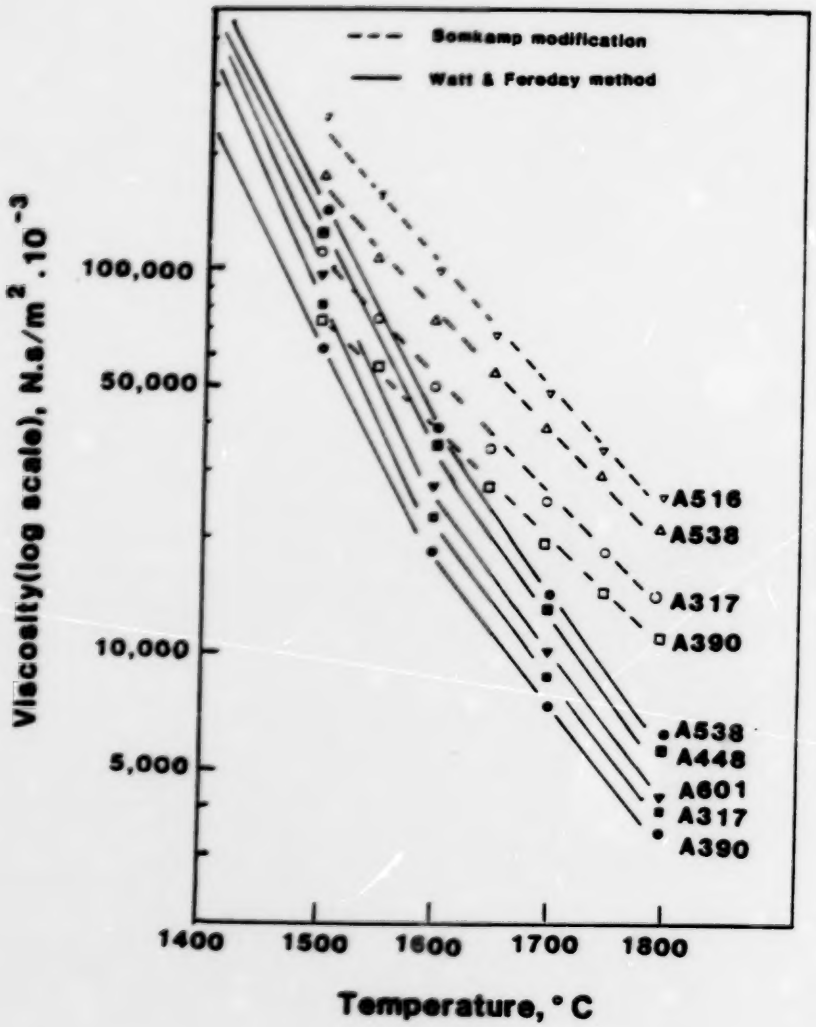

Fig. 13 - Calculated viscosity-temperature relationship for the slag samples.

Table 1 - Chemical compositions of the slag samples

Sample number	Original* number	SiO_2	Al_2O_3	TiO_2	Fe_2O_3	CaO	MgO	Na_2O	K_2O	P_2O_5	LOF
A448	319182	67.58	20.32	0.47	3.49	4.16	1.17	0.87	0.31	0.28	0.5
A516	319682	68.89	19.81	0.45	3.63	4.17	1.12	0.92	0.30	0.24	0.5
A317	320182	63.30	20.02	0.32	4.58	5.43	1.19	2.56	1.61	0.48	0.5
A390	320682	61.70	20.16	0.44	4.98	5.99	1.22	1.86	1.10	0.43	0.5
A538	365182	67.57	22.12	0.70	3.93	2.58	0.90	1.26	0.33	0.34	0.5
A559	365682	67.97	21.13	0.67	4.64	2.46	0.91	1.12	0.34	0.27	0.5
A601	366682	61.76	26.97	1.17	3.92	2.29	0.75	0.71	0.84	0.03	0.5
A202											
S1	S1	44.72	24.25	0.66	4.48	13.78	3.75	2.49	1.38	0.43	0.7
S2	S2	42.43	22.30	0.81	5.81	13.95	3.71	2.69	1.55	0.39	5.49

*Designated sample number by CCRL

Table 2 - Thermal behaviour of the slag samples

Sample no.	Determined by ASTM technique								Determined by heating Microscope		
	Under oxidizing condition, °C				Under reducing condition, °C				In air, C		
	I	S	H	F	I	S	H	F	S	H	F
A448	1322	1407	1435	1455+	1310	1365	1404	1454	1300	1400	1460
A516	1332	1366	1399	1455+	1310	1327	1363	1443	1320	1420	1480
A317	1238	1298	1326	1355	1199	1255	1299	1343	1250	1360	1400
A390	1232	1260	1316	1355	1198	1255	1293	1349	1300	1350	1370
A538	1426	1455+			1404	1454+			1400	1480	1520
A559	1413	1454+			1382	1430	1454		1400	1500	1520
A601	1454+				1454+				1500	1560	1600
S2	1205	1225	1245	1305	1160	1195	1205	1280	1180	1240	1290
A202									1300	1380	1400

Table 3 - Slags samples, origins and types of coals from which they are derived

Sample no.	Original no.	Rank of coal	Coal origin
A538	365182	medium volatile bituminous	British Columbia
A601	366682	medium volatile bituminous	Alberta
A202	20282	medium volatile bituminous	British Columbia
A516	319682	high volatile bituminous	Alberta
A390	320682	subbituminous	Alberta
A317	320182	subbituminous	Alberta
A448	319182	high volatile bituminous	Alberta
S1	S1	lignite (fly ash)	Southern Saskatchewan
S2	S2	lignite (bottom ash)	Southern Saskatchewan
S3	S3	lignite (lagoon ash)	Southern Saskatchewan

Table 4 - Calculated SiO_2/Al_2O_3 and $SiO_2/(Al_2O_3+Fe_2O_3)$ mass ratio for the slag samples

Sample no.	SiO_2/Al_2O_3	$SiO_2/(Al_2O_3 + Fe_2O_3)$
A448	3.32	2.48
A516	3.47	2.94
A317	3.16	2.57
A390	3.06	2.45
A538	3.05	2.59
A601	2.28	1.99
S2	1.90	1.51
S1*	1.84	1.55

*Fly ash from Poplar Generating Station, Saskatchewan.

Table 5 - Calculated base to acid ratio of the coal slags

Sample no.	Base to Acid ratio
A448	0.11
A516	0.12
A317	0.18
A390	0.18
A538	0.09
A601	0.09
S2*	0.42
S1*	0.37

*Fly ash from Poplar Generating Station, Saskatchewan

Table 6 - Chemical analyses of the as-received slags and re-analyses after viscosity runs

	A448		A601	
	Original	Melt	Original	Melt
SiO_2	67.58	66.79	61.76	64.92
Al_2O_3	20.32	20.92	26.97	22.01
TiO_2	0.47	0.73	1.17	0.8
Fe_2O_3	3.49	4.87	3.92	4.99
CaO	4.16	4.51	2.29	2.96
MgO	1.17	0.86	0.75	0.71
Na_2O	0.87	0.63	0.71	0.64
K_2O	0.31	0.83	0.84	1.21
P_2O_5	0.28	0.20	0.30	0.31
BaO		0.01		0.54
SrO		0.01		0.10

Table 7 - A suggested rating scheme for predicting slagging

Slagging category	Temperature, °C
Low	1343
Medium	1260 - 1343
High	1176 - 1260
Severe	1176

Table 8 - Rating of the slags in terms of slagging

Sample no.	Temperature, °C	Slagging category
A448	1377	low
A516	1346	low
A317	1256	medium
A390	1249	medium
S2	1184	high